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TRANSLATION

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JAPANESE PATENT SPECIFICATION

No. NS 110620/74

PROCESS FOR PRODUCTION OF PULVERULENT MALTITOL

Filing date: March 8, 1973

Specification published:(laid open unexamined): October 22, 1974

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Hyogo-ken

PATENT CLAIM

A process for production of pulverulent maltitol, which process comprises concentrating maltitol from about 75% concentration by sub-atmospheric pressure concentrating means, atmospheric pressure falling film concentrating means or the like means to a solid of at least 98%, and grinding said solid; selecting a pulverulent substance which is compatible with foodstuffs

Please Note—Names of Japanese firms, research laboratories and government entities, as translated, are not necessarily identical with the names adopted by such organizations for international contacts. Japanese personal and surnames often permit of several readings and the ones used in this translation are not necessarily the ones preferred by their bearers. Foreign names mentioned in Japanese specifications cannot always be accurately reconstructed.

and remains free-flowing under normal temperature and humidity conditions and admixing said substance to the ground maltitol, which admixture may alternatively take place before the grinding step, by which admixture a barrier is laid up between the characteristically hygroscopic maltitol particles and between the atmosphere and said particles, thereby inhibiting caking of said particles, which barrier has a thickness of 3000  $\mu$  or less.

#### EXPERIMENTAL

(The experiments relate to the seeded and unseeded spray-drying or spray crystallization of maltitol syrups. They purport to show that the production of free-flowing powders is impossible by these methods. This section has not been translated. Translator.)

#### EXAMPLES

In conformity with the invention, ordinary maltitol was concentrated to a solids content of at least 98%. The solid concentrate was crushed into pebbles or flakes and these introduced into a mechanical grinder or pneumatic attrition mill.

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The various coating agents tested, and the proportions by weight in which they were used on maltitol were as follows: 5-7% dextrin, 5-7% lactose, 5-7% dextrose, 5-7% maltose, 0.5-1% CMC, 0.5-1% methylcellulose, 0.5-5% locust bean gum, 0.5-5% carrageenan, 0.5-1% Avicel (microcrystalline cellulose), 0.5-1% calcium phosphate  $\text{Ca}_3(\text{PO}_4)_2$ , 0.1-0.5% silicic anhydride, 0.1-0.5% magnesium silicate, 0.1-0.5% calcium stearate and 0.1-0.5% (? illegible).

In the proportions stated above, the coating agent was mixed with the crushed, solid concentrate or it was introduced in the grinding step. These operations were carried out through the four seasons and their changing atmospheric conditions and in each case the maltitol was recovered as a product with the appearance of crystalline, granulated sugar. (It is not explained how the coating agent segregates itself from the maltitol in the mixing operation and becomes an encapsulating outside layer. Fig. 1 relates to something different, viz. the spray-drying of seeded maltitol. Translator.)

(1) The coating agent consisted of  $\text{SiO}_2$  of 3  $\mu$  particle size. It was introduced in the proportion of

0.48% by weight (including 3.8% moisture content). The coated maltitol particles had a diameter of 600  $\mu$ . The product had an angle of repose of 32°. The grinding step was carried out under atmospheric conditions of 23°C and 75% R.H.

The finished particles were exposed to the same atmospheric conditions as in the grinding step, and moisture absorption measured over a period of time. Results are reflected in graphs 2 and 3. In both graphs the x-axis is the time scale (hours in graph 2 and minutes in graph 3) and the y-axis represents weight gain, in percent. Both graphs indicate a 0.45% weight increase in the samples after about 2 hr exposure to the atmosphere, meaning that caking or lumping has started. In both graphs a very steep rise in the curve to about 0.3% moisture absorption is apparent. This is evidence of rapid initial moisture absorption from the atmosphere due to the hygroscopic nature of maltitol concentrated to a moisture content of 2% or less. Graph 2 shows the more specific results obtained in an experiment in which the state of the atmosphere to which the product was exposed was changed during the holding time. The relative humidity

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was changed from 75% to 54%, with the temperature remaining unchanged.

Graph 4 reflects the flow characteristics, expressed as angle of repose, of the pulverulent product on a time scale of minutes (x-axis). The y-axis represents the angle of repose. It is apparent that the product remains in a free-flowing condition (corresponding to an angle of repose of  $32-40^{\circ}$ ) for about 2 hr. The line A---B marks the entry into the caking zone. The 2 hr period is ample for use of the product. In industrial handling the product can be pneumatically conveyed or carried on a conveyor in 2 hr without giving rise to problems.

This product (100 g) was sealed into a 100x200 mm polyethylene envelope and the latter placed in an atmosphere of  $30^{\circ}\text{C}$  and 80% R.H. for 7 days. The seal was then broken and the free-flowing property of the contents retested in terms of its angle of repose. This was found to have remained unchanged at  $32^{\circ}$ . When a 3 kg load was placed on the envelope for 24 hr the contents failed to agglomerate or cake. However, when this test was carried out on maltitol coated with

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only 0.2%  $\text{SiO}_2$ , about one-third of the envelope contents agglomerated into lumps, although these could be broken up by finger touch.

(2)  $\text{SiO}_2$  of 7  $\mu$  particle size was used in the proportion of 0.5% by weight (including 3.8% moisture). The finished particles had a size of 600  $\mu$  and an angle of repose of  $32^\circ$ . The temperature and relative humidity of the atmosphere in the grinding step were substantially the same as in Example (1). The product was tested in an atmosphere of  $30^\circ\text{C}$  and 80% R.H. for 2 hr. About one-third of the total sample caked up under its own weight during this test, but the lumps broke up when touched. This was, however, not the case when the material was left in the atmosphere for 24 hr. About one-third was firmly caked or lumped up.

(3) Maltitol was coated with  $\text{SiO}_2$  of 100  $\mu$  particle size in the proportion of 0.5% by weight of maltitol. The results were poor: Within 2-3 min. about one-third of the product caked or lumped up.

(4) Maltitol was coated with magnesium silicate (talc) of 3  $\mu$  particle size. The conditions under which the operation was carried out were substantially the same

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as those used in the above examples, and the results were likewise substantially the same. However, in the weighted envelope test about one-third of the product caked or lumped up within 2 hr.

参考文献—林原商(株)パンフレット：マルビット

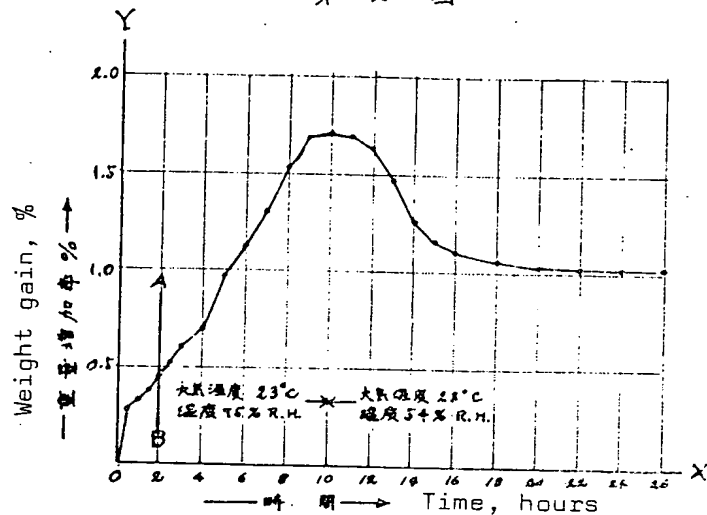
林原生物化学研究所：マルビットの性質と応用について

特開 49-110620 (5)

第 1 図

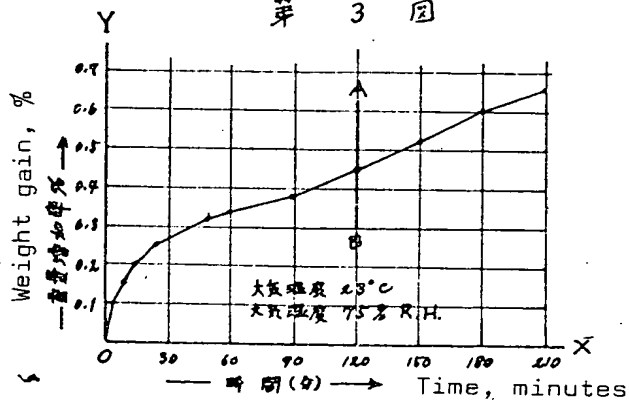


第 2 図

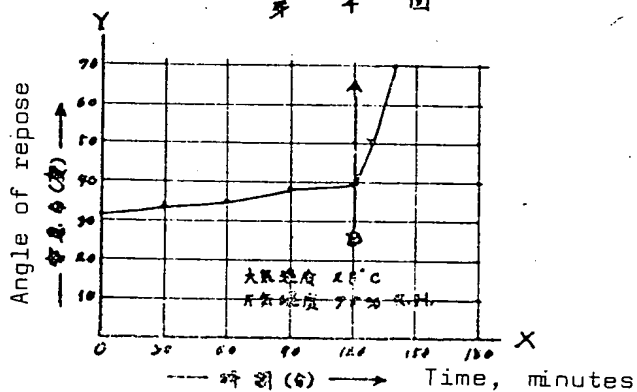


- 23 -

第 3 図



第 4 図





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JAPANESE PATENT SPECIFICATION

No. 49-110620 (1974)

PROCESS FOR PRODUCTION OF PULVERULENT MALTITOL

Filing date: March 8, 1973, No. 48-27305 (Examination requested)

Specification published: unexamined: October 22, 1974

Inventor(s): T. Hiraiwa

Assignee: K. Oyamada

FULL DISCLOSURE OF THE INVENTION

Maltitol is a sugar alcohol derived from a disaccharide obtained by conversion of sweet potato or cornstarch. The compound has recently attracted much attention as a sweetener for low calorie diets. Its sweetness is 85-95% that of cane sugar. The sweetness differs entirely from that of saccharin and the like synthetic sweeteners and is very similar to that of cane sugar. It is a mellow sweetness, free from unpleasant after-taste. Maltitol therefore may be expected to gain a place for itself in medicine and in the food industry. Unfortunately the sugar alcohol both attracts and holds moisture from the atmosphere, is noncrystalline, and even when concentrated to a

supersaturated state, stored at a low temperature or spray-dried, does not crystallize.

For these reasons maltitol is usually made as a 75% syrup (sold under the trade name Marbit) or as pebble-like toffies which present a minimal surface area per given weight unit and are individually wrapped in moisture-proof wrapping material. These are inconvenient to handle, package and store and difficult to transport and ship. Therefore a process for preparing maltitol in pulverulent form would be of great advantage. So far, no practical process has been put forward. This invention relates to a process for production of pulverulent maltitol.

Maltitol syrup (75%) at a temperature of 25° has a viscosity of 1,100 cp. The syrup is colorless and transparent. The viscosity drops sharply with rise in temperature. Maltitol is resistant to high temperatures, both physically and chemically. It has for example no tendency to turn brown. When the syrup is boiled down to 98% solids or more it solidifies to a glassy substance. This stubbornly retains the remaining moisture and in this respect excels sorbitol, glycerol and other polyhydric alcohols used as humectants. About the difficulty of drying and related properties of maltitol the attached pamphlet issued by Hayashibara Shoji and Hayashibara Biochemical Research Institute may be consulted. [Not reproduced with the patent

application, but the graphs may have been taken from the pamphlet.]

When the solidified maltitol comes in contact with humid air it at once absorbs moisture and becomes tacky and soft. It is therefore a strongly hygroscopic and deliquescent substance, much more so than for example crude solar salt. The grinding of maltitol pebbles or flakes would be impossible under the humidity conditions obtaining (in Japan) throughout the year. This is because the particles immediately attract humidity from the atmosphere and mutually coalesce. Particles of 3,000 microns or smaller diameter are difficult to recover. Even when the grinding is carried out under low temperature and low humidity conditions such as 8°/50% R.H. the aggregation of particles cannot be prevented. The powder recovered has substantially no free-flowing properties. When exposed to the atmosphere, the particles begin to lump at once and soon become immobile inside their container. These are the conditions which render the preparation of pulverulent maltitol difficult.

The following is a relevant discussion of general methods of grinding and spray crystallization as applied to maltitol.

(1) Spray Crystallization

When a 75% maltitol syrup is concentrated to a predetermined viscosity and introduced into a spray-drying column in which it is atomized and dried in a hot-air stream at 110-180°,

it is found that it is difficult to dehydrate the syrup droplets to less than 5% water. Even if a water content of 7% were acceptable, ---- (? illegible) and heat energy requirements would remain very substantial. An air temperature of at least 90° is required at the final spray-drying stage. For this reason the spray-dried particles are in a softened state. When the pulverulent product is treated with cooling air at 8°/40% R.H. in the product discharge zone or in the final drying zone with due regard to the dew point, it is possible to lower the discharge temperature to 33°, but a free-flowing powder remains elusive. The building up of a deposit of particles on the inside wall of the discharge zone is a consequence of inadequate drying. Particles which reach the outside and make contact with air immediately form large aggregations and lumps. A free-flowing pulverulent product has never been obtained in this inventor's experiments.

As a great deal of heat is required inside the spray dryer to evaporate the maltitol droplets to substantial dryness, the cooling and solidification of the maltitol particles is no easy matter. The use of both hot air blast and cold air blast zones, the latter at the dew point, has likewise proved a failure, causing the sprayed particles to clog up the hopper zone and the product discharge zone. To sum up, the production of dry, particulate maltitol by spray-drying is an impossibility.

Some specific observations made are the following:

(a) It is practically impossible to lower the moisture content of the particles to less than 5%;

(b) Uneconomically large-sized spray-drying equipment would be required for any continuous drying and cooling operation; and

(c) A pulverulent product with a particle size of more than 100 microns has poor free-flowing properties and its particle porosity renders the powder highly hygroscopic.

(2) Seeded Spray-drying

The syrup was seeded with lactose, dextrin, maltose, etc. in the proportion of 0.5% to 50%.

Compared with unseeded spray-drying, the rate of powder formation appeared a little higher but the proportion deposited on the dryer was ~~was~~ much higher, hence the yield of product available in the collection basket was considerably lower. In a continuous spraying operation only 100 g of powder was recovered from a feed of 25 kg of 75% maltitol syrup (Marbit). Stability of the product to the atmosphere is about the same as by the unseeded method.

The reason why seeded spray-drying produces no crystals is the incompatibility of the seed crystal with the uncrystallizable maltitol. This is schematically shown in Fig. 1 in which the hatched portions are maltitol molecules and the unhatched portions represent seed crystals. As no effective seed encapsulation takes place, the maltitol at the interfaces

in contact with humid air absorbs water which quickly engulfs the seed crystals. The whole turns into sticky particles, and these particles aggregate into lumps.

(3) Granulation by Spray Cooler-granulator

Maltitol syrup was concentrated to 97% solids and heated to 80°. At this temperature the viscosity was 500-600 cp. The concentrate was sprayed through a nozzle under 10 kg/cm<sup>2</sup> pressure in the expectation of producing particles of about 380 microns (40 mesh) diameter, but many particles were joined into strings and could not be completely separated. Many particles were fused with others before cooling. Granulation by this method was therefore impossible.

As it became apparent from the results of the above-cited experiments that maltitol particles, once formed, are unstable in humid atmospheres, this inventor directed his attention to methods of isolating the particles from the environmental influences by coating or by microencapsulation. Long-chain alcohols, edible paraffins, oils and fats, etc. were dissolved in a solvent, the maltitol particles admixed to the solution and recovered with a coating which is dried.

An alternative method would take advantage of the spontaneous dampness and tackiness of maltitol particles due to moisture absorption from the atmosphere and coat the particles

by dusting with a very finely divided barrier substance, but this method, when reduced to practice on an industrial scale would seem to call for the installation of highly complex equipment.

Microencapsulation would appear to be impractical with present-day methods and in any event would be of economically doubtful soundness.

The following are examples of the practice of this invention as set out in the patent claim. [Separately translated.]

TRANSLATOR'S NOTE

This patent specification is somewhat unsatisfactory in that it omits particulars of the procedures and equipment used in the preparation of the coated particles.

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WPI Acc No: 75-16687W/197510

Free-flowing maltitol prepn - liq. maltitol evapd. and pulverized with  
powd. additives

Patent Assignee: K OYAMADA (OYAM-I)

Number of Countries: 001 Number of Patents: 001

Patent Family:

Patent No	Kind	Date	Applicat No	Kind	Date	Main IPC	Week
JP 49110620	A	19741022					197510 B

Priority Applications (No Type Date): JP 7327305 A 19730308

Abstract (Basic): JP 49110620 A

Liq. maltitol is evapd. to >98% concn. and pulverised with  
nontoxic, powdery additives of good fluidity. The surface coating  
prevents adhesion of particles. In an example, maltitol contg.<2% H2O  
was pulverised with 0.48% SiO2 of particle size 3 mu. The product of  
av. particle size 600 mu retained fluidity over 2 hr at 23 degrees and  
75% relative humidity.

Derwent Class: D17